

Characterization of Si Surface by SEM-SXES using Low Incident Voltage

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Soft X-Ray Spectrometry (SXES) with high energy resolution is a useful method for chemical bonding state analysis by detecting the change in spectrum shape. In recent years, a soft X-ray spectrometer, which can be mounted on electron microscope, has been developed. [1] Especially, if mounted onto an FE-SEM, chemical bonding state analysis of bulk material surface can be performed using a low voltage electron beam. Moreover, depth analysis of chemical bonding state is possible by changing the incident voltage. [2] In this study, we have tested SEM-SXES analysis using low incident voltages, for analysis of the damaged layer on cross section of Si wafer, to evaluate the change in crystallinity of silicon by analyzing the chemical bonding state. Results of this study are reported below.

The system for this study is a Schottky FE-SEM, JEOL JSM-7900F, in combination with the soft X-ray spectrometer, JEOL SS-94000SXES. In-lens Schottky Plus FEG mounted on JSM-7900F is uniquely integrated electron gun and condenser lens system. This integration enables a more efficient utilization of the electrons from the electron gun. Therefore, it is possible to provide large probe currents for SXES analysis at low accelerating voltages. The energy range of SS-94000SXES is from 50 eV to 210eV, and the energy resolution is 0.3 eV (Al-L Fermi edge of metallic aluminum).

Samples for this study are: cross section of a cleaved Si wafer (sample A), cross section of a Si wafer prepared by 8kV Ar⁺ ion beam (sample B), and sample B after additional fine mill by 4kV Ar⁺ ion beam (sample C). To check the spectrum shape change at the damaged layer close to the sample surface formed by Ar⁺ ion beam irradiation, each of the cross sections of Si wafer were SXES analyzed at incident voltage of 2 keV, 1keV, and 500 eV. Furthermore, we carried out Electron Backscattered Diffraction (EBSD) analysis using TSL OIM for each of the cross sections of Si wafer, to check the quality of EBSD pattern.

Fig.1 shows the EBSD patterns of each samples, taken at 15 kV accelerating voltage, 10 nA probe current. EBSD pattern obtained from Sample A (Fig.1-a) gives the clearest EBSD pattern of the samples. EBSD pattern of sample B (Fig.1-b) is a low intensity pattern due to the damaged layer close to surface, formed by 8kV Ar⁺ ion beam irradiation. On the other hand, the quality of the EBSD pattern obtained from sample C (Fig.1-c) is improved in comparison with sample B, the damaged layer was reduced by fine milling.

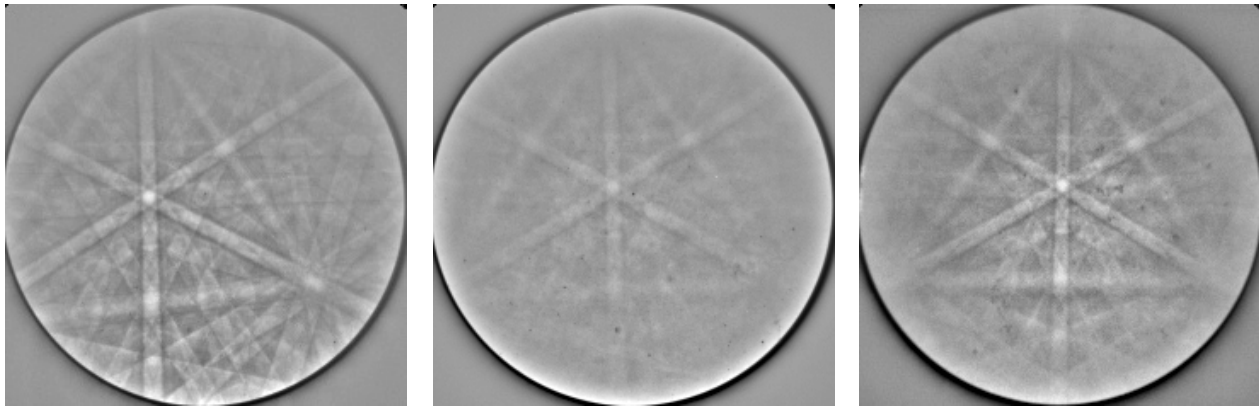
Fig.2 shows the spectra of Si L_{2, 3}-M₁, obtained from each sample by SEM-SXES analysis. When comparing the incident voltages of 2 keV, 1 keV and 500 eV, there are no spectrum shape change observed on sample A (Fig.2-a). However, from the results of sample B (Fig.2.b), the spectrum shape changes with lower incident voltage, thus confirming that the chemical bonding state was altered. Moreover, the result of sample C (Fig.2.c) shows a more gradual change of the spectrum shape. These results are consistent with EBSD pattern qualities.

These results lead to the possibility of a new evaluation technique for changes in material surface crystallinity by chemical bonding state analysis using SEM-SXES at low incident voltages.

References:

[1] M. Terauchi *et al*, Journal of Electron Microscopy **61** (2012), p. 1.

[2] H. Takahashi *et al*, Microscopy and Microanalysis **22(S3)** (2016), p.422.

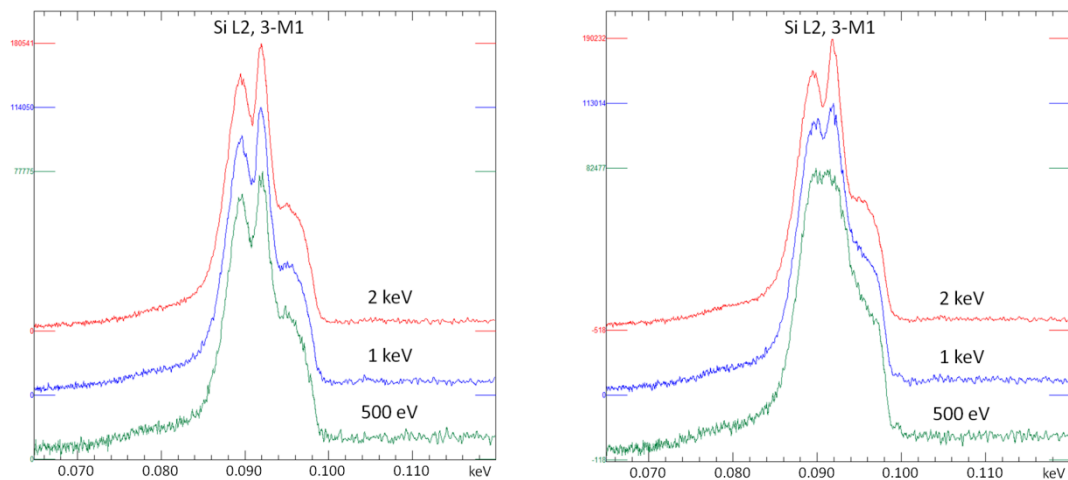


(a) Cleaving

(b) 8 kV Ar⁺ ion beam milling

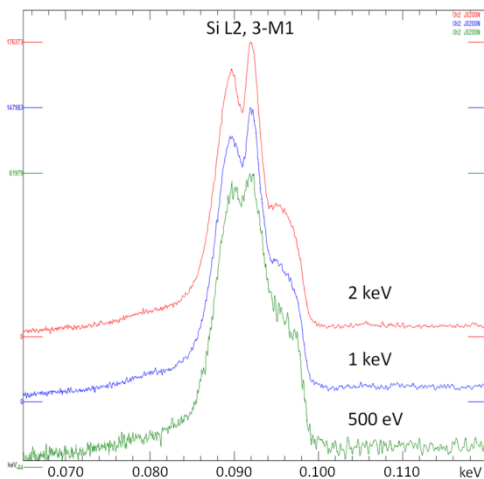
(c) 8 kV Ar⁺ ion beam milling and 4 kV fine milling

Figure 1. EBSD Pattern of each Si wafer cross section



(a) Cleaving (Sample A)

(b) 8 kV Ar⁺ ion beam milling (Sample B)



(c) 8 kV Ar⁺ ion beam milling and 4 kV fine milling (Sample C)

Figure 2. SXES results: Si L_{2,3}-M₁ Spectra of each Si wafer cross section